

(19)



Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

EP 1 061 124 A1

(12)

## EUROPEAN PATENT APPLICATION

(43) Date of publication:

20.12.2000 Bulletin 2000/51

(51) Int. Cl.<sup>7</sup>: C11D 3/50, C11D 17/04

(21) Application number: 00112230.8

(22) Date of filing: 07.06.2000

(84) Designated Contracting States:

AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU  
MC NL PT SE

Designated Extension States:

AL LT LV MK RO SI

(30) Priority: 15.06.1999 US 139278 P

(71) Applicant: Givaudan SA  
1214 Vernier-Genève (CH)

(72) Inventors:

- McGee, Thomas  
95690 Nesles la Vallee (FR)

• Purzycki, Kenneth Leo

Lake Parsippany, New Jersey 07054 (US)

• Sgaramella, Richard P.

Hoboken, New Jersey 07030 (US)

(74) Representative:

Patentanwälte

Schaad, Balass, Menzl & Partner AG

Dufourstrasse 101

Postfach

8034 Zürich (CH)

### (54) Method for preparing fragrance products

(57) The present invention relates to a process for reducing fragrance loss in a substrate. This process includes preparing a protected fragrance matrix that contains a perfume, an optional fixative and a solid absorbent. The process further includes combining a substrate conditioner with the fragrance matrix and a substrate. Products, especially fabric softener sheets, made from these processes are also provided.

EP 1 061 124 A1

BEST AVAILABLE COPY

## Description

**[0001]** The present invention relates to substrates having prolonged fragrance. More particularly, the present invention provides compositions and methods for reducing the loss of perfume during manufacture.

**[0002]** Fabric softener sheets typically include a substrate, a fabric conditioner base and a fragrance. The fabric conditioner base is used to impart a benefit to a fabric treated therewith. Such a benefit may include, for example, softness, reduced static of dried articles, easier ironing, etc. The fragrance is used to impart a pleasant aroma to the dried fabric.

**[0003]** In use, these fabric softener sheets are typically placed in a conventional dryer together with fabric to be dried, e.g., wet laundry. During the drying process, fragrance and other benefits are transferred from the fabric softener sheet to the fabric being dried.

**[0004]** Conventionally, a fabric softener sheet is manufactured by coating a mixture of fabric conditioner base and fragrance onto a substrate. Typically, fabric softener bases are solid mixtures that must be melted prior to use. In practice, fabric conditioner bases are melted in a holding tank at about 70°C to 90°C then fragrance is added. Depending on the speed of coating, the conditioner base and fragrance may remain at these elevated temperatures for 4 to 8 hours. Fabric softener sheets are coated by continuously passing a substrate, e.g., a polyester sheet material, at high speed through a coating tank that is also held at 70°C to 90°C. The coated sheet material is then passed through a series of cooled rollers and a cooling tower to reduce the sheet temperature to about 30°C.

**[0005]** Fragrance losses occur at various points during the conventional fabric sheet manufacturing process; these losses in total may be up to 30 percent to 45 percent.

**[0006]** Encapsulates, such as cyclodextrin compositions as outlined in U.S. Patent No. 5,348,667, have been used to reduce perfume loss during the manufacturing process. Such encapsulation technology, however, is expensive to make and use, and is therefore not cost effective in a large-scale commercial setting.

**[0007]** Absorbents have been used to carry lipophilic materials such as fragrance in the manufacture of certain products. For example, it is known that solids can adsorb oily materials, such as, perfume. Such adsorbent solids have been reportedly used as carpet cleaning powders (U.S. Patent No. 5,783,543 and U.S. Patent No. 5,286,400), in oil adsorbent products (U.S. Patent No. 4,537,877 and U.S. Patent No. 5,763,083), in detergent powders (U.S. Patent No. 5,656,584), in detergent liquids (U.S. Patent No. 4,983,422 and U.S. Patent No. 4,209,417), in detergent bars (EP 816484), and in liquid softeners (U.S. Patent No. 4,954,285).

**[0008]** Accordingly, one object of the present invention is to provide a process for producing a fabric softener sheet which process significantly reduces the loss of the perfume in the fragrance matrix during the manufacture of such a sheet compared to conventional processes.

**[0009]** Another object of the invention is to provide a substrate having prolonged fragrance compared to conventionally prepared substrates.

**[0010]** Another object of the invention is to provide an efficient, cost-effective method for reducing the loss of perfume during the manufacture of fabric softener sheets.

**[0011]** The present invention is directed to meeting these and other objects.

**[0012]** The present invention provides a process for reducing fragrance loss during the manufacturing of fragranced substrates. This process includes providing a protected fragrance matrix that contains a perfume, a solid absorbent and optionally a fixative. In this process, a fabric conditioner base is further provided. A substrate is then combined with the fragrance matrix and the fabric conditioner base. A substrate treated in this manner retains more of the fragrance compared to conventionally prepared substrates.

**[0013]** The present invention also provides a substrate impregnated with a fragrance matrix having prolonged organoleptic activity. This substrate is made by the process of forming a protected fragrance matrix that includes a perfume, an optional fixative and a solid absorbent. The fragrance matrix is then combined with a fabric conditioner base. The fragrance matrix and fabric conditioner base are then applied to the substrate either together as a mixture or sequentially. In this process, the fragrance matrix is combined with the fabric conditioner base immediately prior to application of the mixture to the substrate. Alternatively, the fragrance matrix is applied to the substrate immediately after the fabric conditioner base.

**[0014]** The present invention provides a low cost method of reducing the loss of perfume during the manufacture of, e.g., a fabric softener sheet. This process includes the addition of a protected, i.e., an enrobed perfume contained within a fragrance matrix to a fabric conditioner base prior to application of the fabric conditioner base onto or into a substrate or during one of the rolling stages of a conventional fabric softener sheet manufacturing process. By using the process of the present invention, it is easier to dose, i.e., apply the fragrance to the substrate during the manufacturing process when the perfume is in a solid carrier form.

**[0015]** Thus, the process of the present invention includes (1) preparing a fragrance matrix including an enrobed, i.e., a protected perfume, an optional fixative and a solid absorbent; and (2) combining the fragrance matrix with a fabric conditioner base and a substrate.

**[0016]** As used herein, the term "fabric softener sheet" includes a substrate, a fabric softener base and a fragrance matrix. For purposes of the present invention, the term "substrate" is intended to mean any material that acts as a delivery vehicle for the fragrance matrix and fabric conditioner base. Thus, in the present invention, the substrate must be able to retain in its interior or on its surface a sufficient quantity of the fabric conditioner base and fragrance matrix required to deliver the desired performance (prolonged fragrance) to the fabric, e.g., a fabric softener sheet. Typically, the substrate is in the form of a sheet, although other forms may be used. The substrate may be a synthetic or natural fiber that is woven, knitted or extruded. Non-limiting examples of materials that may be used as a substrate in the present invention include cotton, Rayon, polyester, regenerated cellulose, and the like.

**[0017]** As used herein, the phrases "fragrance matrix" or "protected fragrance matrix" mean that at least the most volatile parts of a perfume are protected or enrobed by a solid absorbent and optionally a fixative to reduce the amount of loss of fragrance during the manufacturing process of a substrate through evaporation of the perfume volatiles.

**[0018]** In the present invention, the protected fragrance matrix is said to "enrobe" the perfume. Thus, as used herein, the terms "enrobe," "enrobed" or "enrobement" refer to the sequestration of the perfume in the protected fragrance matrix to reduce the loss of fragrance during the manufacture of fragranced substrates, e.g., fabric softener sheets, and to prolong the effect of a fragrance on substrates coated with such an enrobed perfume.

**[0019]** The protected fragrance matrix is preferably in the form of a flowable powder. Thus, the ratio of perfume, solid absorbents and fixative are carefully controlled to maintain the consistency of a flowable powder. In this embodiment, the perfume-to-solid absorbent ratio is between about 1:20 to about 2:5, preferably about 1:5. The level of perfume, however, will depend upon the final composition of the substrate. The fragrance, in turn, may be present in the protected fragrance matrix in an amount of from about 10% to about 50% (wt/wt), preferably, in an amount of from or between about 20% to about 40% (wt/wt). On the substrate, the fragrance level will be about 1% to about 6% (wt/wt).

**[0020]** The flowable powder may be dosed into, for example, a molten fabric conditioner base such as a softener or an antistatic system. Preferably, the powder is mixed into the fabric conditioner base immediately prior to application of the mixture to the substrate. Alternatively, the powder is applied to a substrate previously coated with the fabric conditioner base at a subsequent processing step, such as at one of the rolling stations during the manufacture of a fabric softener sheet.

**[0021]** As used herein, the term "immediately" is intended to mean that the fragrance matrix is added to the fabric conditioner base just prior to application to the substrate. Typically, the fragrance matrix is added 1-20 minutes prior to application to the substrate, preferably 2-10 minutes prior to application to the substrate; most preferably the fragrance matrix is dosed at the rolling stage directly onto the substrate coated with the fabric conditioner base.

**[0022]** In the present invention, the protected fragrance matrix may contain all or part of the perfume. If part of the perfume is protected, i.e., enrobed, then it is usual to select the most volatile components to be enrobed.

**[0023]** For example, in the present invention, the protected fragrance matrix is formed into a powder by mixing the perfume with an absorbent solid and an optional fixative. Absorbent solids that may be used in accordance with the present invention include, for example, clays, silicas, Celites® (ICN Biomedicals, Inc., Aurora, OH, USA.), zeolites, metal salts, including for example phosphates, cellulose, such as methyl cellulose, starches, carbonates, such as sodium bicarbonate, borates, such as sodium borate, sulfates, such as sodium sulfate, water-soluble polymers, borax and mixtures thereof.

**[0024]** For purposes of the present invention, the term "perfume" is intended to mean a mixture of a single or complex mixture of aromatic chemicals which have been formulated to give an aesthetically pleasing smell to a substrate, such as for example, a fabric softener sheet. As used herein, "aroma chemicals" are intended to mean chemicals that have an odor. There are many chemical classes which fall within the meaning of the phrase "aroma chemicals" as used herein, including for example, hydrocarbons, alcohols, aldehydes, ketones, lactones, esters. These chemical classes are described in S. Arctander Perfume Flavors and Chemicals, Vols. 1 and 2, Arctander, NJ, USA.

**[0025]** The perfume may also contain small amounts of other additives, such as solvents, preservatives, antioxidants, UV screening agents and the like. The fragrance matrix may also include organoleptic components, such as for example, other well-known fragrance ingredients.

**[0026]** Preferably, a fixative is incorporated into the fragrance matrix of the present invention. The fixative preferably is a high molecular weight, low melting solid composition that may be mixed into a powder. Examples of suitable fixatives for use in the present invention include polyethylene glycol, Glycerox (Croda Inc., Parsippany, NJ, USA.), mineral oil and mixtures thereof. In accordance with the present invention, the molecular weight of the fixative may vary between about 400 to 20,000 daltons, preferably between about 2,000 to 10,000 daltons. Other fixatives having a similar viscosity and melting point to polyethylene glycol are also contemplated by the present invention. The level of fixative useful in accordance with the present invention is between about 1% and about 40% (wt).

**[0027]** The solids and the liquids set forth above are combined by, for example mixing in a suitable mixer until the blend is flowable so that it is easy to apply (i.e., dose) to a substrate.

**[0028]** The present invention also includes a fabric conditioner base. For purposes of the present invention, the fabric conditioner base includes a mixture of ingredients that impart a desired property to the fabric, such as for example,

softness, no static cling, reduced creasing, easier ironing, and the like. These properties are achieved using, for example, cationic, zwitterionic, and non-ionic softeners, soaps and quaternary compounds, as well as other fatty materials alone or in combination. Other examples of suitable fabric softener bases may be found in U.S. Patent No. 3,686,025.

[0029] Other optional components may also be added to the fabric conditioner base to further impart various desired characteristics to the substrate, so long as these ingredients do not significantly interfere with the reduction of fragrance loss during manufacturing of substrates. Such optional components include, for example, antistatic agent(s), anti dye-transfer agent(s), soil release agent(s), and the like.

[0030] In the process according to the present invention, the substrate may be coated at several carefully timed points and according to a specific sequence of events to achieve the desired fragrance effect. For example, in one embodiment, the substrate may be coated by combining the fragrance matrix with the fabric conditioner base to form a mixture immediately prior to applying the mixture to the substrate. Alternatively, the substrate is coated by applying the fabric conditioner base thereto to form a substrate-fabric conditioner base composition. Thereafter, the fragrance matrix is applied to the coated substrate.

[0031] For purposes of the present invention, the fragrance matrix and/or fabric conditioner base is applied to the substrate using any conventional technique suited for the particular application, such as, for example, spraying, coating, dipping and the like. Using these and other techniques, the fragrance matrix and fabric conditioner base are applied to the substrate as a coating. Alternatively, the fragrance matrix and fabric conditioner are impregnated throughout the substrate, i.e., are dispersed within the interstitial matrix of the substrate as well as its surface.

[0032] Preferably, the fabric conditioner base and fragrance matrix are dispersed throughout the substrate to a uniform thickness of about 0.1 to about 2 mm to evenly distribute the fragrance throughout the entire substrate.

[0033] Another embodiment of the present invention is a substrate impregnated with a fragrance matrix having prolonged organoleptic activity. This substrate is made by the process which includes (a) forming a protected fragrance matrix as set forth previously with a perfume, a fixative and a solid absorbent; and (b) mixing the fragrance matrix with the fabric conditioner base immediately prior to applying the mixture to the substrate or applying the fragrance matrix to the substrate immediately after the fabric conditioner base is applied to the substrate.

[0034] Thus, in one embodiment, a mixture of the fragrance matrix and fabric conditioner base is applied to the substrate. In this embodiment, the fragrance matrix is added to the fabric conditioner immediately prior to application of the mixture to the substrate. Alternatively, the fabric conditioner base and fragrance matrix are applied to the substrate sequentially. In this embodiment, the fabric conditioner is applied to the substrate first, immediately followed by the fragrance matrix.

[0035] The fixative according to this embodiment is selected from high molecular weight, low melting point compositions such as for example, polyethylene glycol, Glycerox, mineral oil and mixtures thereof.

[0036] The following examples are set forth to illustrate the processes and compositions of the present invention. These examples are provided for purposes of illustration only and are not intended to be limiting in any sense.

## EXAMPLES

### Example 1

#### Enrobed Composition

[0037] An enrobed, i.e., protected fragrance matrix was made by combining the ingredients as set forth below:

Component	%
Perfume Oil (Givaudan Roure) Fragrance PNF217FPN	20
Polyethylene Glycol 4000 (Union Carbide)	1
Bentonite H (Whitaker, Clark & Daniels)	10
Valfor® 100 Aluminosilicate Zeolite (PQ Corp.)	69

**Example 2****Comparison of Fragrance Retention of Enrobed Composition vs. Control**

- 5 [0038] A comparison between a fabric softener sheet made using a conventional process and a fabric softener sheet made according to the processes of the present invention are set forth below.

**Control**

- 10 [0039] A conventional fabric softener base, Varisoft 136-100 (Witco, Inc. OH) was heated to 80° C in a convection oven. 48 grams of this composition was placed in a pre-heated beaker and 2 grams of the perfume oil PNF217FPN (fragrance level = 4%) was added to the beaker with agitation. The mixture was held at 80° C for 4 hours to simulate production and then applied to a commercially available polyester softener sheet in a pilot plant using a coating machine (Talbot Engineering Corp.).

**Enrobed Composition**

- 15 [0040] Varisoft 136-100 (Wites Corp., Dublin, OH, USA) was heated to 80° C and 40 grains thereof was placed in a pre-heated beaker as described above. 10 grams of the protected fragrance matrix (20% PNF217FPN = 4% fragrance) according to Example 1 was added to the Varisoft with mixing immediately prior to application of the mixture onto a fabric softener sheet in a pilot plant.

- 20 [0041] On cooling, the fragrance level in the control and enrobed softener sheets was determined by extraction and analysis by gas chromatography/mass spectrometry. The results are shown below:

25

Percent of theoretical fragrance level	
Control Sheet	28.8
Enrobed Sheet	43.2

30

- 35 [0042] As the data above indicates, perfume loss in the pilot plant was reduced from 71% to 57% using the late addition of a protected fragrance matrix (Enrobed Sheet) compared to the control sheet.

**Example 3****Enrobed vs. Unprotected Compositions in Open/Closed Systems**

- 40 [0043] The manufacture of fabric softener sheets requires that a melted fabric softener base and a fragrance be held in an open/closed vessel at 80°C (176°F) for up to 4 hours. To demonstrate the ability of the present enrobed processes to reduce fragrance loss, two samples were set up as follows:

**Control (Non Protected Perfume)**

- 45 [0044] 48 grams of Varisoft 136-100 was melted in a convection-draft oven at 80°C (176°F). 2 grams of PNF217FPN (Fragrance level = 4%) was added to the Varisoft composition and held at 80°C for 4 hours in an open beaker.

**Enrobed Composition**

- 50 [0045] 40 grams of Varisoft 136-100 base was melted in a convection-draft oven at 80°C (176°F) and 10 grams of a protected fragrance matrix according to the present invention (20% PNF217FPN = 4% fragrance level) from Example 1 was added to the base. The mixture was held at 80°C for 4 hours in an open beaker.

- 55 [0046] Both samples were extracted and analyzed. The results are shown in Table 2.

Table 2

Fragrance Loss From Base After 4 Hours in an Open Vessel at 80°C			
Control		Enrobed Composition	
% Fragrance	% Loss	% Fragrance	% Loss
2.2	45%	2.9	27%

[0047] As Table 2 indicates, when the enrobed composition of the present invention is added to the melted base and left for four hours, the amount of fragrance loss is reduced by about 50% as compared to the control.

[0048] The above-referenced experiment was repeated but in a closed vessel rather than an open beaker. The results are shown in Table 3 below:

Table 3

Fragrance Loss From Base After 4 Hours in a Covered Vessel at 80°C			
Control		Enrobed Composition	
% Fragrance	% Loss	% Fragrance	% Loss
2.92	27%	3.32	17%

[0049] As Table 3 indicates, even in a closed vessel the Enrobed composition of the present invention reduced the amount of fragrance loss by about 50% compared to the control.

#### Example 4

##### Effect of Prolonged Heating on Enrobed vs. Unprotected Compositions

[0050] A commercially available fragrance PNF217FPN (Givaudan Roure) was used to determine what effect prolonged heating would have on a conventionally prepared fabric conditioner base and a fabric conditioner base containing a protected fragrance matrix according to the present invention.

[0051] Control and enrobed samples as described in Example 3 were heated and maintained at 80°C for 8 hours. Samples were taken every 2 hours, extracted and analyzed by GC-MS. The data obtained is set forth in Table 4 below:

Table 4

Fragrance Level Summary					
Sample	Time (Hours)				
	0hrs	2hrs	4hrs	6hrs	8hrs
Conventional	3.6	3.3	3.2	3.1	2.9
Enrobed (Protected)	3.9	3.9	3.7	3.7	3.5

[0052] As Table 4 indicates, the protected fragrance matrix does not evaporate as much as the neat oil and affords protection from the heated environment.

[0053] Table 5 shows the amount of fragrance lost from the system. After 8 hours at 80°C, the enrobed fragrance matrix still contains twice as much fragrance as the conventional formulation.

Table 5

Fragrance Loss Summary					
Sample	Time (Hours)				
	0hrs	2hrs	4hrs	6hrs	8hrs
Conventional	10%	17.5%	20%	22.5%	27%
Enrobed (Protected)	2.5%	2.5%	7.5%	7.5%	12.5%

[0054] As Table 5 demonstrates, the use of the protected fragrance matrix, according to the present invention, does protect the fragrance to the extent that even after prolonged heating losses are less than those experienced under conventional production conditions.

#### Example 5

##### Enrobed vs. Unprotected Compositions

[0055] The manufacture of fabric softener sheets requires that the melted fabric softener base and fragrance be held in an open/closed vessel at about 80°C (176°F) and continuously added to a coating pan which is also maintained at about 80°C (176°F). To demonstrate the ability of Enrobed compositions of the present invention to reduce fragrance loss, two samples were set up as follows:

##### **Sample 1 (Control)**

[0056] 48 grams of Varisoft 136-100 base was melted in a convection-draft oven at 80°C (176°F). 2 grams of PNF217FPN (Fragrance level = 4%) was immediately added to the base and fabric softener sheets prepared.

##### **Sample 2 (Enrobed Composition)**

[0057] 40 grams of Varisoft 136-100 base was melted in a convection-draft oven at 80°C (176°F) and added to a coating pan. When the temperature was stable, 10 grams of the Enrobed composition of the present invention containing PNF217FPN (20% fragrance = 4% fragrance level) from Example 1 was added to the base and fabric softener sheets were prepared.

[0058] Fabric softener sheets from both experiments were extracted and analyzed. The results are shown in Table 6.

Table 6

Fragrance Loss From Fabric Softener Sheets With and Without Enrobement			
Control		Enrobed	
% Fragrance	% Loss	% Fragrance	% Loss
2.86	29%	3.55	11.3%

[0059] As Table 6 indicates, the enrobed composition of the present invention reduced the amount of fragrance loss by about 60% compared to the control.

#### Example 5

[0060] One of the highest levels of fragrance loss during the manufacture of fabric softener sheets occurs when the sheets are cooled. Adding fragrance near the end of the process or after coating should significantly reduce fragrance loss.

[0061] To test this hypothesis, a protected fragrance matrix according to the present invention was sprinkled uni-

formly onto the surface of warm sheets freshly coated with Varisoft fabric softener base. The sheets were then cooled, extracted, and analyzed.

**[0062]** Table 7 compares the fragrance loss of conventionally prepared sheets to sheets prepared using the enrobed composition of Example 1 after coating.

Table 7

Fragrance Loss From Fabric Softener Sheets With and Without Protected Fragrance Matrixes			
Normal Coating Process		Enrobed Fragrance	
% Fragrance	% Loss	% Fragrance	% Loss
2.4	40%	3.88	3%

**[0063]** As Table 7 indicates, adding the Enrobed composition (Enrobed Fragrance) from Example 1 to warm fabric softener base-coated sheets significantly reduces fragrance loss by about 90% compared to conventional coating processes.

#### **Example 6**

##### **Comparison of Effect of Different Fixatives in Enrobed Composition**

**[0064]** Three different samples were prepared as in Example 1 except that the following fixatives were substituted for the polyethylene glycol 4,000 recited therein: (a) polyethylene glycol with a molecular weight of 20,000 daltons; (b) Glycerox (a triglyceride mixture) with a molecular weight of about 2,000 daltons; and (c) mineral oil, with a molecular weight of approximately 400 daltons.

##### **Dryer Sheet Preparation:**

##### **Immediate Application:**

**[0065]** Varisoft 136-100 was used as the fabric conditioner base. It was heated to 80°C in a convection oven. For each of the three samples, 40g of Varisoft were placed into a pre-heated beaker. 10g of the enrobed composition as prepared in Example 1 was added with mixing. 1.5g of the molten mixture was evenly spread on a 1g blank woven polyester dryer sheet.

##### **4 Hour Heat/Hold Application:**

**[0066]** Varisoft was heated to 80°C in a convection oven. 40g of the Varisoft was weighed into a pre-heated beaker and 10g of the enrobed fragrance matrix was added, mixed and held at 80°C for 4 hours and then applied onto a sheet using the same procedure as above.

**[0067]** The fragrance level of each sheet was determined by Soxhlet extraction followed by GC-MS analysis. The results are shown in Table 8 below.

Table 8

Sample	Fragrance (%)	Loss (%)
PEG 20000	3.94	1.5
Glycerox	3.42	14.5
Mineral Oil	3.51	12.3
Conventional-4 hour heat/hold	3.18	21
Mineral Oil-4 hour heat/hold	3.08	23
Conventional Dryer Sheet	2.4	40



[0068] As the data in Table 8 indicate, each of the three fixatives used with the protected fragrance matrix of the present invention reduce fragrance loss compared to a conventionally prepared dryer sheet. The higher molecular weight (PEG 20,000) sample provides the best effect of the three fixatives tested exhibiting a percent fragrance loss of only 1.5%.

[0069] A similar fragrance protection was also evident in the 4-hour heat/hold samples as well. The immediate application of the protected fragrance matrix continues to be the best variant. However, even the heat/held dryer sheets showed a 53% improvement in fragrance loss when compared to the conventionally prepared product.

#### Example 7

#### Effect of Absorbents on Protected Fragrance Matrix

[0070] Enrobement or protection of the perfume in the fragrance matrix may be broken up into two parts: the fixative and the absorbent. In this example, the absorbent effects, particularly the replacement of clay and zeolite absorbents with several other absorbents is shown. The fragrance absorption capacity of the dry blends was also tested and is presented below.

[0071] Alternative absorbents to zeolite and clay were tested as follows; starch, methyl cellulose, sodium sulfate, sodium bicarbonate and sodium borate. Blends of these alternative absorbents were prepared at 50% with zeolite, 75% with clay, and at 100% with no zeolite or clay.

[0072] Results are listed in Tables 9-11. The sodium bicarbonate and sodium borate blended 50/50 with zeolite and 20% fragrance formed a pasty wet mass (sodium bicarbonate) and large moist clumps (sodium borate). Replacing the zeolite with clay had the same results. These results indicate that sodium bicarbonate and sodium borate are not optimal absorbents.

[0073] The starch, methyl cellulose and sodium sulfate all performed well either blended with zeolite or clay. They remained free flowing and easily absorbed up to 40% fragrance before showing any signs of clumping.

[0074] Thus, the data in Tables 9-11 indicate that several materials are suitable for use as absorbents in the present fragrance matrix either in place of or blended with zeolite and clay. As Tables 9-11 also indicate, the absorbent blends have a capacity of up to about 40% fragrance while remaining free flowing and homogeneous.

[0075] Tables 9-11 also demonstrate that various carriers and fragrances may be used in the present fragrance matrix by balancing each component to produce a free-flowing powder that is easily handled and applied to e.g., fabric softener sheets. Thus, any combination of a fragrance and a suitable carrier that results in a free flowing composition that is easily handled and dosed is encompassed by the present invention.

Table 9

Absorbent Summary : 50/50 Sample/Zeolite Blend				
Sample	Fragrance Amount		Free Flowing Powder	
	20%	30%	20%	30%
Starch	Good	No Good	Free Flowing	Wet Paste- NG
Methyl Cellulose	Good	Good	Free Flowing	Free Flowing
Sodium Sulfate	Good	No Good	Free Flowing	Wet Clumps
Sodium Bicarbonate	No Good	N/A	Wet Paste	N/A
Sodium Borate	No Good	N/A	Wet Clumps	N/A

Table 10

Absorbent Summary: 75/25 Sample/Clay Blend				
Sample	Fragrance Amount		Free Flowing Powder	
	20%	30%	20%	30%
Starch	Good	Good	Free Flowing	Free Flowing

Table 10 (continued)

Absorbent Summary: 75/25 Sample/Clay Blend				
Sample	Fragrance Amount		Free Flowing Powder	
	20%	30%	20%	30%
Methyl Cellulose	Good	Good	Free Flowing	Free Flowing
Sodium Sulfate	Good	Good	Free Flowing	Free Flowing
Sodium Bicarbonate	No Good	N/A	Wet Paste	N/A
Sodium Borate	No Good	N/A	Wet Clumps	N/A

Table 11

Absorbent Summary : 100% Sample				
Sample	Fragrance Amount		Free Flowing Powder	
	20%	30%	20%	30%
Starch	Good	No Good	Free Flowing	Wet Paste- NG
Methyl Cellulose	Good	No Good	Free Flowing	Free Flowing
Sodium Sulfate	Good	No Good	Free Flowing	Wet Clumps
Sodium Bicarbonate	No Good	N/A	Wet Paste	N/A
Sodium Borate	No Good	N/A	Wet Clumps	N/A

**Example 8****Comparison of the Fragrance Retention of Conventional and Enrobed Dryer Sheets**

**[0076]** Fabric softener sheets were prepared by adding the appropriate amounts of protected fragrance matrix, as set forth in Example 1, on to a blank polyester sheet dosed with the applicable amount of active as set forth in Example 1. This process simulates post dosing and was prepared at a fragrance level of 4%, and compared to a conventionally prepared sheet.

**[0077]** Headspace GC-MS measurements were made from conventionally prepared fabric softener sheets, from fabric softener sheets prepared according to the present invention, and from towels that had been washed, then dried, with the respective fabric softener sheets. The results set forth in Table 12 indicate that the sheets containing the protected or enrobed fragrance, i.e., the fabric softener sheets made according to the present invention, retain far more fragrance and deposit much more fragrance on to towels in the dryer compared to commercially available alternatives.

**[0078]** Moreover, in an olfactive assessment the 4% protected fragrance matrix-containing dryer sheets were unanimously ranked higher for fragrance strength and hedonics.

Table 12

Headspace Comparison: Enrobed Dryer Sheets	
Sample	Fragrance: Ng/Liter
Conventional Market Sheet	11,000
4.0% Enrobed Sheet	28,000
Dried Towel Conventional Sheet	41
Dried Towel 4.0% Enrobed	192

[0079] Thus, the data in Table 1 indicate that protecting the perfume component of a fragrance matrix by enrobing it does provide protection to the fragrance during high temperature preparation of dryer sheets.

[0080] The invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention and all such modifications are intended to be included within the scope of the following claims.

### Claims

1. A process for reducing fragrance loss in a substrate comprising preparing a protected fragrance matrix comprising a perfume and a solid absorbent, and combining the fragrance matrix with a fabric conditioner base and the substrate.
2. A process according to claim 1 wherein the fragrance matrix is combined with the fabric conditioner base to form a mixture, and immediately applying the mixture to the substrate.
3. A process according to claim 1 wherein the fabric conditioner base is combined with the substrate to form a substrate-fabric conditioner base composition and then the fragrance matrix is combined with the substrate-fabric conditioner base composition.
4. A process according to claim 1 further comprising dispersing the fabric conditioner base and the fragrance matrix throughout the substrate to a uniform thickness of about 0.1 to about 2.0 mm.
5. A process according to claim 1 wherein the solid absorbent is selected from the group consisting of clays, celites, silicas, zeolites, metal salts, celluloses, starches, carbonates, borates, sulfates, water-soluble polymers, Borax and mixtures thereof.
6. A process according to claim 1 wherein the solid absorbent is selected from the group consisting of zeolites, clays, methyl celluloses, sodium sulfates, starches, bicarbonates and mixtures thereof.
7. A process according to claim 1 wherein the fragrance matrix comprises a low melting point, high molecular weight fixative.
8. A process according to claim 7 wherein the fixative is selected from the group consisting of polyethylene glycol, Glycerol, mineral oil and mixtures thereof.
9. A process according to claim 8 wherein the polyethylene glycol has a molecular weight from about 400 to 20,000 daltons.
10. A process according to claim 8 wherein the polyethylene glycol has a molecular weight from about 2,000 to 10,000 daltons.
11. A process according to claim 1 wherein the fabric conditioner base comprises a fabric softener selected from the group consisting of cationic softeners, zwitterionic softeners, non-ionic softeners and mixtures thereof.
12. A process according to claim 11 wherein the fabric conditioner base further comprises a component selected from the group consisting of an antistatic agent, an anti dye-transfer agent, a soil release agent and mixtures thereof.
13. A process according to claim 1 wherein the fragrance matrix comprises additional fragrance components.
14. A process according to claim 1 wherein the fragrance matrix comprises perfume in an amount of from about 10% to about 50%.
15. A process according to claim 1 wherein the fragrance matrix comprises perfume in an amount of from about 20% to about 40%.
16. A process according to claim 1 wherein the substrate is a synthetic or natural material.
17. A process according to claim 16 wherein the substrate is selected from the group consisting of polyester, Rayon,

cellulose and mixtures thereof.

18. A process according to claim 1 wherein the substrate is a sheet.

19. A process according to claim 1 wherein the perfume to solid absorbent ratio is from 1:20 to about 2:5.

20. A process according to claim 1 wherein the perfume to solid absorbent ratio is from about 1:5.

21. A substrate made from the process of claim 1.

22. A fabric treated with a substrate made by the process of claim 1.

23. A substrate coated or impregnated with a fragrance matrix having prolonged organoleptic activity prepared by the process comprising:

(a) forming a protected fragrance matrix comprising a perfume, a fixative and a solid absorbent;

(b) applying the fragrance matrix and a fabric conditioner base to the substrate, wherein the fragrance matrix is mixed with the fabric conditioner base immediately prior to application of the mixture to the substrate or the fragrance matrix is applied to the fabric conditioner base immediately after the fabric conditioner base is applied to the substrate.

24. The substrate of claim 23 wherein the perfume to solid absorbent ratio is from about 1:20 to about 2:5.

25. The substrate of claim 23 wherein the perfume to solid absorbent ratio is from about 1:5.

26. The substrate of claim 23 wherein the fixative is a low melting point, high molecular weight composition.

27. The substrate of claim 23 wherein the fixative is selected from the group consisting of polyethylene glycol, Glycerox, mineral oil and mixtures thereof.



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 00 11 2230

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
X	US 4 073 996 A (BEDENK WILLIAM THOMAS ET AL) 14 February 1978 (1978-02-14)	1-6, 11-19, 21,22 20,23-25	C11D3/50 C11D17/04
A	* claims 1-11,13-18 * * example 2 * * column 2, line 40 - column 4, line 65 * * column 7, line 52 - column 8, line 32 * * column 9, line 51 - column 11, line 42 * * column 13, line 14 - column 14, line 54 * ---		
X	EP 0 536 942 A (UNILEVER) 14 April 1993 (1993-04-14)	1,2,4-6, 11-14, 16,18-22 15,17, 23-25	
A	* claims 1-5,8,9 * * example 9 * * page 3, line 5 - line 48 * * page 4, line 24 - line 33 * * page 5, line 8 - line 24 * ---		
D,X	US 4 954 285 A (NICHOLSON ALYCE E ET AL) 4 September 1990 (1990-09-04)	1,2,4,5, 11,14-22 3,6,12, 13,23-25	TECHNICAL FIELDS SEARCHED (Int.Cl.7) C11D
A	* claims 1-4,10,16-18 * * examples 1,2,9 * * column 3, line 1 - column 5, line 20 * ---		
D,X	US 5 348 667 A (BORCHER SR THOMAS A ET AL) 20 September 1994 (1994-09-20)	1,2,4, 11,14, 16-19, 21,22	
A	* claims * * example 1 * * column 3, line 63 - column 5, line 19 *  * column 8, line 25 - column 11, line 9 * --- -/--	3,12,13, 15,20, 23-25	
The present search report has been drawn up for all claims			
Place of search <b>THE HAGUE</b>		Date of completion of the search <b>21 September 2000</b>	Examiner <b>Neys, P</b>
<p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			

EPO FORM 1503 03.82 (P04C01)



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 00 11 2230

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
D, A	EP 0 816 484 A (GIVAUDAN ROURE INT) 7 January 1998 (1998-01-07)  * claims 1-8,10 * * examples * * page 2, line 9 - line 19 * * page 2, line 49 - page 3, line 32 * ---	1,5-10, 13-15, 19,20, 23-27	
A	CH 541 655 A (COLGATE PALMOLIVE CO) 31 October 1973 (1973-10-31) * claims I, I3-I6, I9-II2, II, III3, III5, III6 * * * examples 4-6 * * column 1, line 49 - column 2, line 26 * * column 14, line 32 - line 52 * -----	1,4,11, 12,16-18	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int.Cl.7)
Place of search	Date of completion of the search	Examiner	
THE HAGUE	21 September 2000	Neys, P	
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 00 11 2230

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

21-09-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4073996 A	14-02-1978	CA 1090507 A	02-12-1980
		JP 1377920 C	08-05-1987
		JP 52124997 A	20-10-1977
		JP 61037384 B	23-08-1986
EP 0536942 A	14-04-1993	AU 2616192 A	08-04-1993
		BR 9203854 A	27-04-1993
		CA 2079242 A	03-04-1993
		FI 924414 A	03-04-1993
		JP 5209189 A	20-08-1993
		KR 9510006 B	04-09-1995
		US 5336665 A	09-08-1994
		ZA 9207595 A	05-04-1994
US 4954285 A	04-09-1990	DE 68927526 D	23-01-1997
		DE 68927526 T	03-07-1997
		EP 0332260 A	13-09-1989
US 5348667 A	20-09-1994	CA 2173436 A	20-04-1995
		EP 0722485 A	24-07-1996
		WO 9510594 A	20-04-1995
EP 0816484 A	07-01-1998	AU 2616697 A	15-01-1998
		BR 9703689 A	27-10-1998
		JP 10060482 A	03-03-1998
		SG 55330 A	21-12-1998
		ZA 9705339 A	10-09-1998
CH 541655 A	31-10-1973	AU 3394971 A	05-04-1973
		BE 773935 A	31-01-1972
		DE 2150586 A	27-04-1972
		DK 43780 A,B,	01-02-1980
		DK 43880 A	01-02-1980
		DK 43980 A	01-02-1980
		DK 147080 B	02-04-1984
		FR 2124010 A	15-09-1972
		GB 1383749 A	12-02-1974
		GB 1383750 A	12-02-1974
		GB 1383748 A	12-02-1974
		IT 944760 B	20-04-1973
		NL 7114461 A	24-04-1972
		SE 7501655 A	14-02-1975
		US 4014105 A	29-03-1977
		ZA 7106518 A	30-05-1973

EPO FORM P459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☒ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**